1-14-92
E-6398

NASA Technical Memorandum 104524

An Optical Method for Determining Level in Two-Phase Cryogenic Fluids

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January 1992

NASA

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INTRODUCTION

Slush hydrogen (SLH_2) is a potential fuel for use in future space vehicles (refs. 1 and 2). In general, slush is a mixture of solid and liquid of the same element at the triple point. This mixture is of variable density and is usually described in units of solid fraction, or quality, on a mass basis.

The metering of slush represents a challenge for several reasons. First, the slush may not be homogeneous, especially under quiescent conditions, because the solid particles settle. In settled slush, there are two levels to be measured: the liquid level and the slush level. For liquid level, a number of techniques show promise for slush applications, including capacitance methods, superconducting methods, time domain reflectometry and magneto-strictive techniques (refs. 3 to 5). But few techniques are also suitable for determining the slush level, or the location of the slush-liquid interface. The technique described in this paper has the ability to measure liquid-gas and liquid-slush interfaces, at the same time. This ability is considered a great advantage (ref. 6).

The second major difficulty in metering slush hydrogen is the low temperature of the hydrogen triple point (13.8 K) (ref. 7). Most electronic devices will not operate at this temperature, which limits their use in instruments at this temperature.

Because of cost, time, and safety considerations, preliminary sensor concept studies were performed in slush nitrogen (SLN_2) rather than SLH_2. The triple point pressure and temperature for SLN_2 are both significantly higher than those for SLH_2. In addition, since SLN_2 is inert, the safety considerations are less stringent than they would be for SLH_2. Of course, there is no guarantee that the two media would behave similarly enough to allow us great confidence in determining the ability of the sensor to operate in both environments. However, we felt that "proof of concept" type tests would be valid in SLN_2. If the concepts are demonstrated in SLN_2, then plans would be made to test the techniques in SLH_2.

While generating slush nitrogen, we observed that the solid-liquid mixture went from transparent to translucent at relatively low solid fractions (fig. 1). This effect became more pronounced as the solid fraction increased. In addition, the settled slush exhibited a clear demarcation between liquid and slush. We thought that an optical attenuation method would be a way to easily measure both the liquid level and the slush level inside the dewar.

In addition to level detection, we felt that an optical attenuation technique would be a candidate for the measurement of quality, or solid fraction. A successful measurement of this type could lead to an inexpensive bulk density measurement system. The conversion from solid fraction to bulk density would be trivial because the cryogen is held at the triple point where solid and liquid densities are known. However, since we had no independent density measurement, our investigation into solid-fraction measurement has been limited to qualitative tests.

The bulk of our experimental work was performed in 'aged' slush. When the slush is newly generated, the particles appear to be agglomerates of very small subparticles loosely attached. As the slush ages, the particles undergo a transformation. The irregularly shaped particles fill in with solid to form a more smoothly spherical

particle. The major portion of this change occurs in the first 5 hr of aging. For slush generators with a substantial heat leak, such as ours, the aging process is accelerated significantly (ref. 8).

SETUP

To investigate the optical attenuation technique for both level detection, and solid fraction measurement, the same experimental setup was used. This rig is shown in figure 2. The slush generator consists of a pair of nested dewars. The outer dewar, containing liquid nitrogen (LN_2), is open to the atmosphere. The inner dewar, also containing LN_2, is sealed with a manifold plate. The manifold plate has pressure ports for LN_2 fill, pressure relief, and other functions.

The slush is generated by using a Venturi pump to lower the pressure in the inner dewar to the nitrogen triple point. As the pressure drops, some of the LN_2 is evaporated into the gaseous phase to maintain the vapor pressure consistent with the LN_2 temperature. The vaporization of the LN_2 draws heat from the surrounding liquid, thus lowering the liquid temperature. As the pressure nears the triple point the LN_2 in the dewar is reduced to the triple point temperature. This is the basis for the "freeze-thaw" method (ref. 9). The lowering of the temperature to the triple point was inferred from the fact that we were generating solid particles of nitrogen, and the mixture was at the triple point pressure, as read from the pressure gage shown in figure 2.

As the liquid at the surface reached the triple point, a layer of solid nitrogen formed at the liquid-gas interface. The Venturi pump is a relatively weak pump; which means that the cycling of pressure normally associated with the freeze-thaw method would be extremely time consuming. Instead of cycling to partially melt the solid at the liquid-gas surface, we used a stirrer to break the solid layer into small chunks which settled to the bottom of the dewar without the necessity of cycling the pressure. The stirrer had the added advantage of agitating the slush to provide a more nearly uniform mixture. As the solid surface was broken, more liquid solidified and was in turn broken up. Without constant stirring, stratification occurred, and equilibrium was achieved through large bubble formation in the warmer liquid at the bottom. By stirring the mixture, we allowed the entire volume to approach the triple point at the same pace.

By closely monitoring and adjusting the pumping rate, a near continuous slush generation was achieved. With our generator we took approximately 2 hr to generate 2.8 liters of SLN_2 from 3.5 liters of normal boiling point LN_2. The loss of volume is caused partially by the increase in density of the mixture, and partially by the evaporation of nitrogen during production.

While investigating the generation of slush nitrogen, we discovered some curious effects. For example, when a light beam such as a laser was directed into the slush, a characteristic beam divergence angle could be distinctly viewed, as is shown in figure 3. Further, the material with the higher solid fraction will produce a higher divergence angle, as can be seen from the larger angle in the lower half of the refracted beam of figure 3.

Viewed directly across from the light source, the refracted beam appears as an elliptical spot. The elliptical shape is an artifact of viewing the beam crosswise through a cylinder. In other words, based upon our observations, a slush mixture of higher density would generate a larger divergence angle, and a bigger spot, than a slush mixture of lower solid fraction. For this reason, we attempted a limited correlation between spot size of the laser beam and solid fraction. To truly investigate this phenomenon, an independent measure of density is required.

The level detector used for this study consisted of a 5-mW helium-neon laser, as the directed light source, and an optical power meter as the receiver. The laser was positioned at one side of the slush generator, diametrically across from the power meter (fig. 4). The laser and meter were aligned by maximizing the power transmitted to the meter. The dewars were mounted on a positioning table between them. We considered it easier to align the optics and move the dewars rather than the instrument. This setup can be seen in figure 5.

The level measurement detailed here was taken after the slush was generated and aged for 1 hr. The starting point of the level detection tests was an arbitrary point located approximately 800 mm from the bottom of the inner dewar. As the test was conducted, the signal from the power meter was sent to the vertical axis input of an xy recorder. The horizontal axis input was time. This axis was converted to length (in millimeters) by a conversion using the (constant) velocity of the table. A typical output of this type is shown in figure 6.

The second portion of our experiment was the measurement of spot size, to be correlated with a rough estimate of solid fraction derived from pressure, temperature, and aging characteristics of the slush. We felt that we could discern at least two clearly separate solid fractions, to be described below, during the course of our investigation. We then prepared to measure the spot size under varying conditions to determine what differences, if any, would appear.

In order to measure the spot size of the laser beam through the slush, we replaced the optical power meter (fig. 4) with a 35-mm SLR camera using a 55-mm f/2.8 lens. The aperture was set to f/8, with a shutter speed of 1/125th sec. The lens was focussed on the inner surface of the inner dewar. An example output of the resultant spot photograph is shown in figure 7. The vertical lines shown in the upper right corner of the photograph are reflections from the flash attachment used to illuminate the ruler that was used for depth measurement. This ruler can be seen (out of focus) in the upper left hand corner of the photograph. This particular photograph was taken with the spot at the bottom of the inside dewar after 1 hr of aging. The slush was being stirred while this photograph was taken.

The photographic data points for this test were taken at three different locations, under varying conditions, as shown in figure 8. The sequence was as follows: We positioned the camera 50 mm below the liquid-gas interface and photographed the spot during slush generation. After the slush was generated, we aged the slush for 1 hr, and positioned the camera 50 mm below the resultant settled slush-liquid line (slush level 1). This became the second photograph. We then stirred the slush with the camera positioned at the same location and took the third photograph. The slush was then allowed to settle for 10 min, to approximately slush level 2, and we positioned the camera at a location 600 mm below the slush level 1. The fourth photograph was taken at this position. Finally, we stirred the slush a second time, and captured the resultant spot, at the same location. This became photograph number 5. Although these by no means constitute a complete set of solid fractions, we believed that these five cases would give us at least two solid fractions to measure. These photographs were then processed off-line to provide a less subjective measurement of spot size.

RESULTS

Figure 6 depicts a typical level detector output. The power passed through the gas is fairly uniform and high (the left side of the curve). As the probe volume passed throughout the liquid-gas interface, there was a sharp drop in transmitted power. In this case, because of nitrogen ice buildup on the inner dewar surface, the power curve did not recover as quickly as one would like. Through the liquid, the power was high, but not as high as in the gas phase. The small fluctuations in both the gas and liquid phases are primarily caused by imperfections in the glass which make up the dewars. The larger fluctuations in the liquid phase are caused by impurities suspended in the LN_2. We believe that the bulk of these impurities are water ice particles which were introduced at the time that the dewar was filled with LN_2.

At the slush-liquid interface there was another sharp drop in transmitted power, and the power transmitted in the slush was uniformly low (the right side of the curve). Volumes represented by this curve are 2.76 liters of triple point slush, and 0.40 liter of liquid above the interface. Figure 6 was obtained in settled slush, so the slush-liquid interface is well defined. During slush generation, since there is no well-defined interface, no method of measurement will provide one.

On the other hand, the determination of spot size from the photographs was significantly more involved. Each spot size negative was enlarged to an 8×10 photo and then digitized so that the central portion filled a 240×240 pixel file. The total result of this image magnification is that a 1-mm square drawn on the inner dewar glass would take up 1 pixel. The center position of each spot was found, using a two-dimensional gaussian curve fit. The spots were all normalized to the center intensity, and the average radial profile of each spot was plotted in figure 9.

Several interesting points can be made about the plots of spot size. During slush generation, the spot was the largest. This, we believe, was caused by the characteristics of the unaged slush. The particles of newly generated slush are of a highly nonuniform shape, with many small dendrites. These nonuniformities afford many more refraction opportunities for the light, causing a larger spot size. This is analogous to having a larger number of much smaller particles. Visually, the unaged slush has the appearance of frosted glass.

Our dewar arrangement had a significant heat leak. The net result of this was the acceleration of the aging process. As the slush ages, the particles becomes more spherical. There are fewer reflecting and refracting surfaces, and the slush takes on the appearance of closely packed glass beads. The spot size decreases for slush that is aged for 1 hr. The difference between these first two curves in figure 9 is caused by this difference in shape of the particles in the two cases.

Stratification occurs during the aging process. Heat leaks cause the liquid at the top to be significantly warmer than the triple point slush lower down. The index difference in the liquid was visible as the slush was stirred after aging. The index difference indicates a temperature difference of about 10 K. Stirring the slush restored equilibrium, but at a lower solid fraction. The warmer liquid melted some solid as it returned to the triple point. The lower solid fraction was reflected in the smaller spot size for the two cases in which the slush was being stirred. Finally, after the slush settled, it stands to reason that the highest solid fraction would be at the bottom of the dewar, and this was where we found the largest spot.

Figure 9 thus reduces to three distinct spot sizes. The smallest spot sizes occurring in aged slush that is being stirred. The intermediate spot size is found in the settled, aged slush near the liquid slush interface. The largest spots are found under two conditions: during slush generation when there is a relatively high number of reflection and refraction surfaces, and at the bottom of the dewar of aged, settled slush. The larger spot size measured during the generation phase poses problems both from a theoretical and operational standpoint for the use of this technique in the determination of slush solid fraction.

CONCLUSIONS

The optical attenuation level detector would work extremely well for settled slush in cases where the experimenter has optical access to the material. Problems, such as glass imperfections, impurities, and nitrogen ice buildup at the liquid surface, encountered in our experiment can be obviated by using an intrusive probe and a better technique for filling the slush generator with liquid nitrogen. This level detection method, however, is unable to determine a definitive slush level during slush generation or during mixing. But this inability is due to the ill-defined slush-liquid interface, not to any inherent fault in the technique.

One method of achieving an intrusive optical probe is to use fiber optics to pipe the source light from the laser and the refracted light to the detector. Some initial work is already being done to identify candidate fibers for use in cryogenic situations (ref. 10).

Finally, we are convinced that the results of the preliminary solid fraction measurement indicate that further study is necessary. As a minimum, correlation between spot size and density needs to be evaluated to determine if this method could conceivably result in a viable instrument. Further research in this area, however requires an independent measurement of density, or solid fraction of the slush, as well as a rudimentary image processing capability.

The major drawback to spot size imaging is the inability to determine whether the larger spots are caused by a larger number of refracting surfaces, as in unaged slush, or by a higher overall density as in aged slush. It may turn out that this technique will prove useful only in aged slush. Further work is needed to resolve this difficulty.

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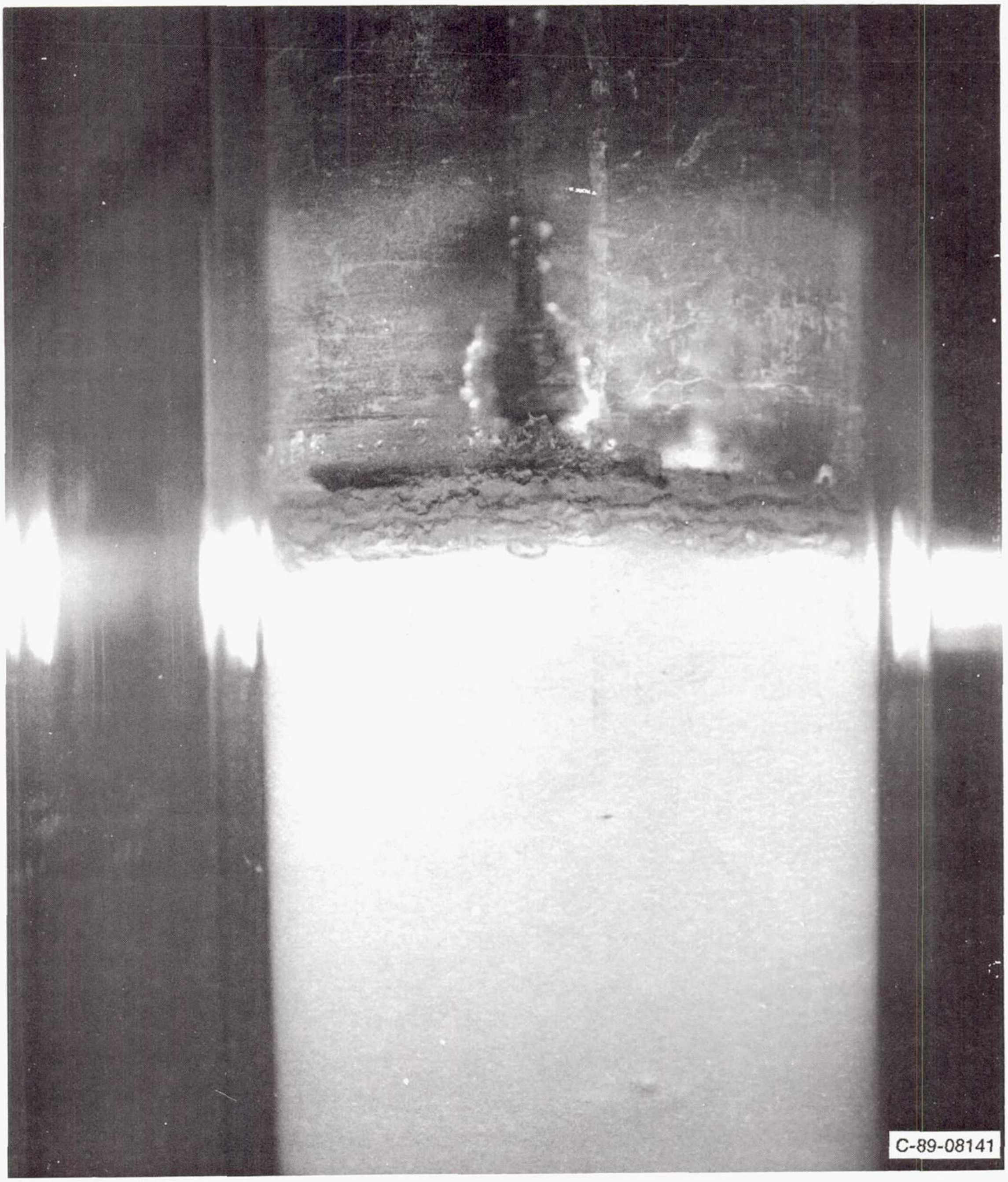

Figure 1.—Slush generation in progress showing translucency of liquid-solid mixture.

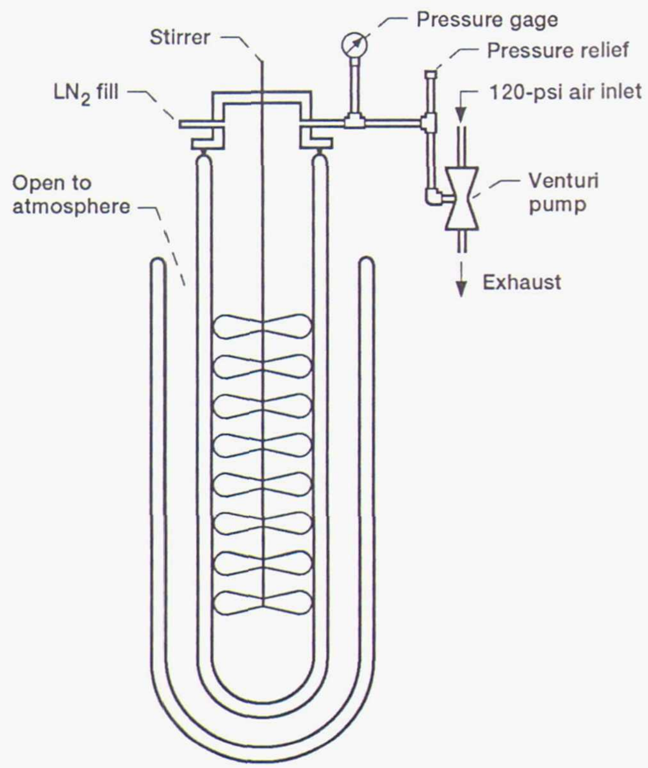

Figure 2.—Slush generator experimental setup.

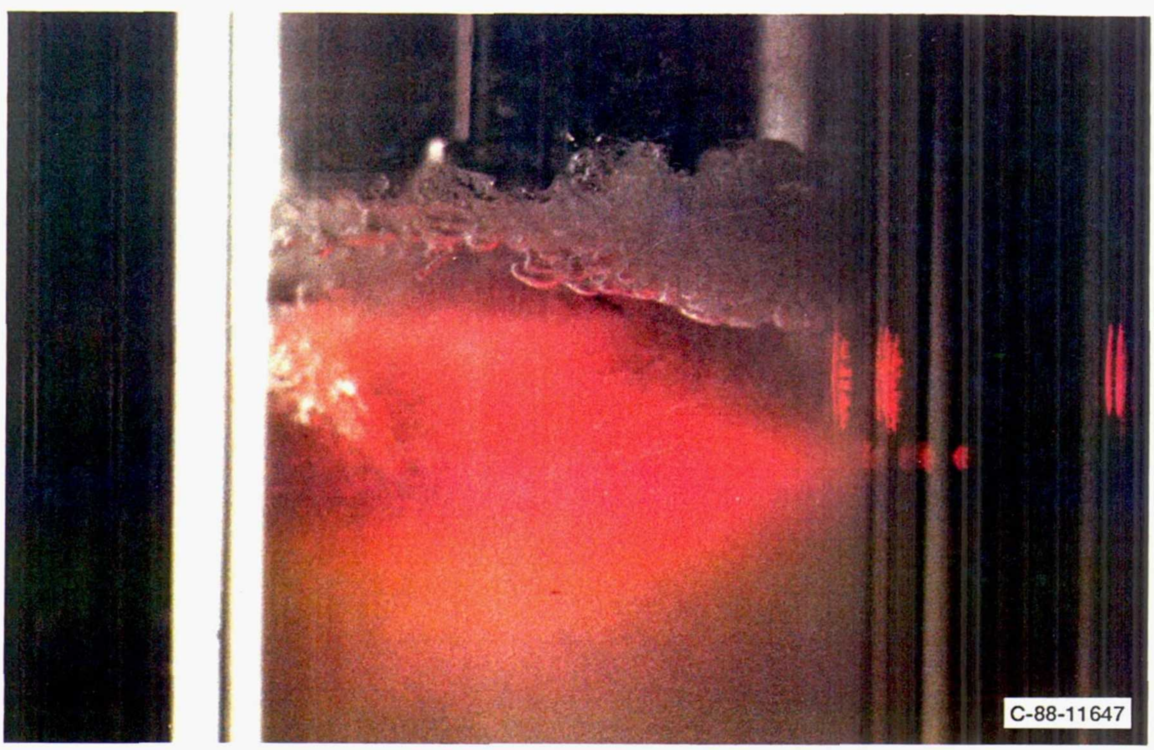

Figure 3.—Characteristic beam divergence angle.

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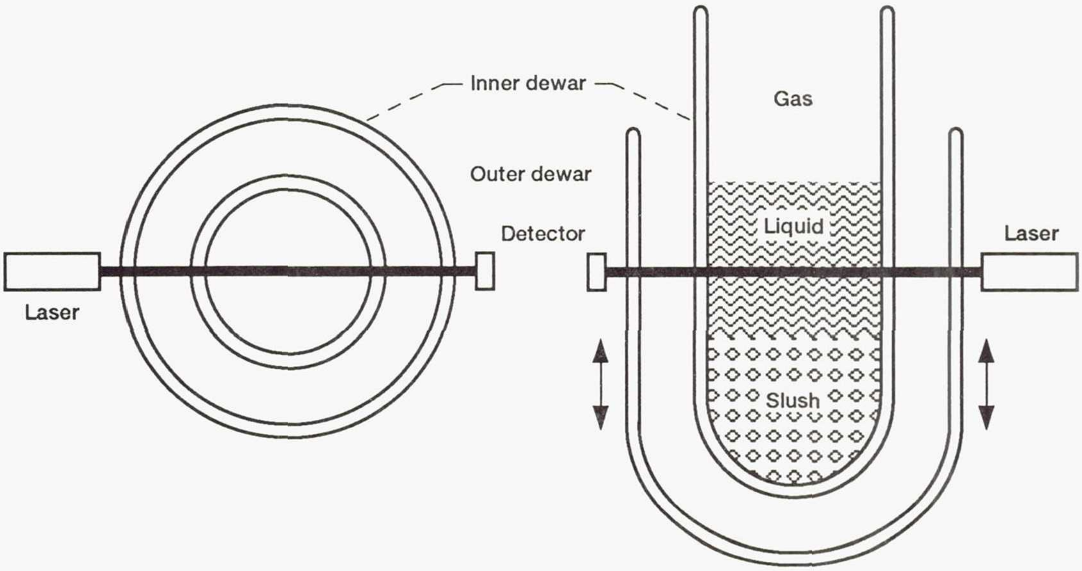

Figure 4.—Instrument location for level detector.

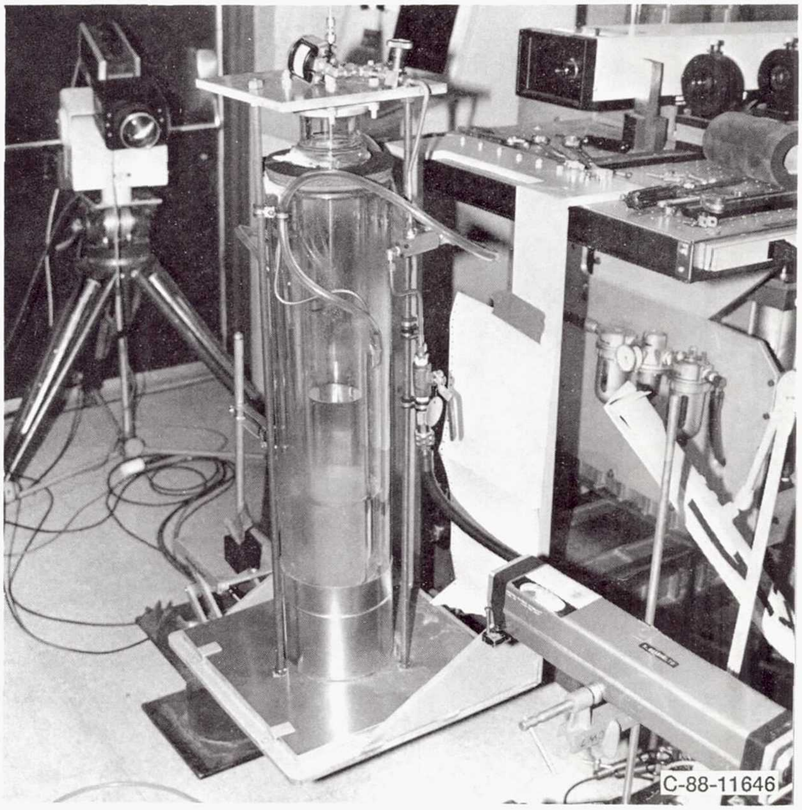

Figure 5.—The slush generator mounted on positioning system.

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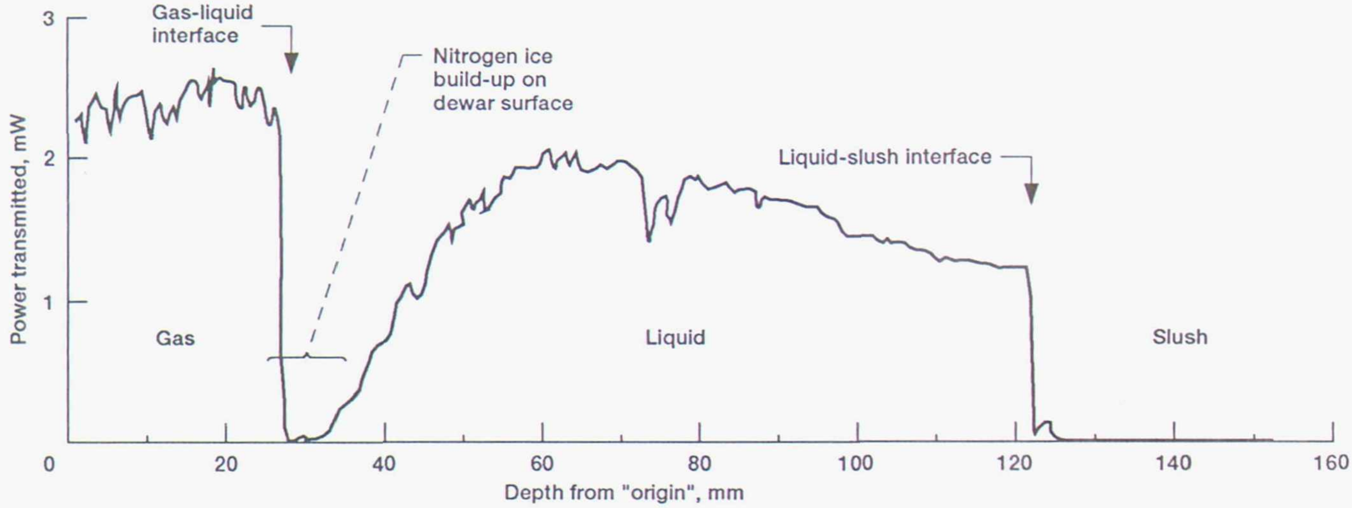

Figure 6.—Level detector sample run showing attenuation as function of the medium 5-mW He-Ne laser passed through both dewars.

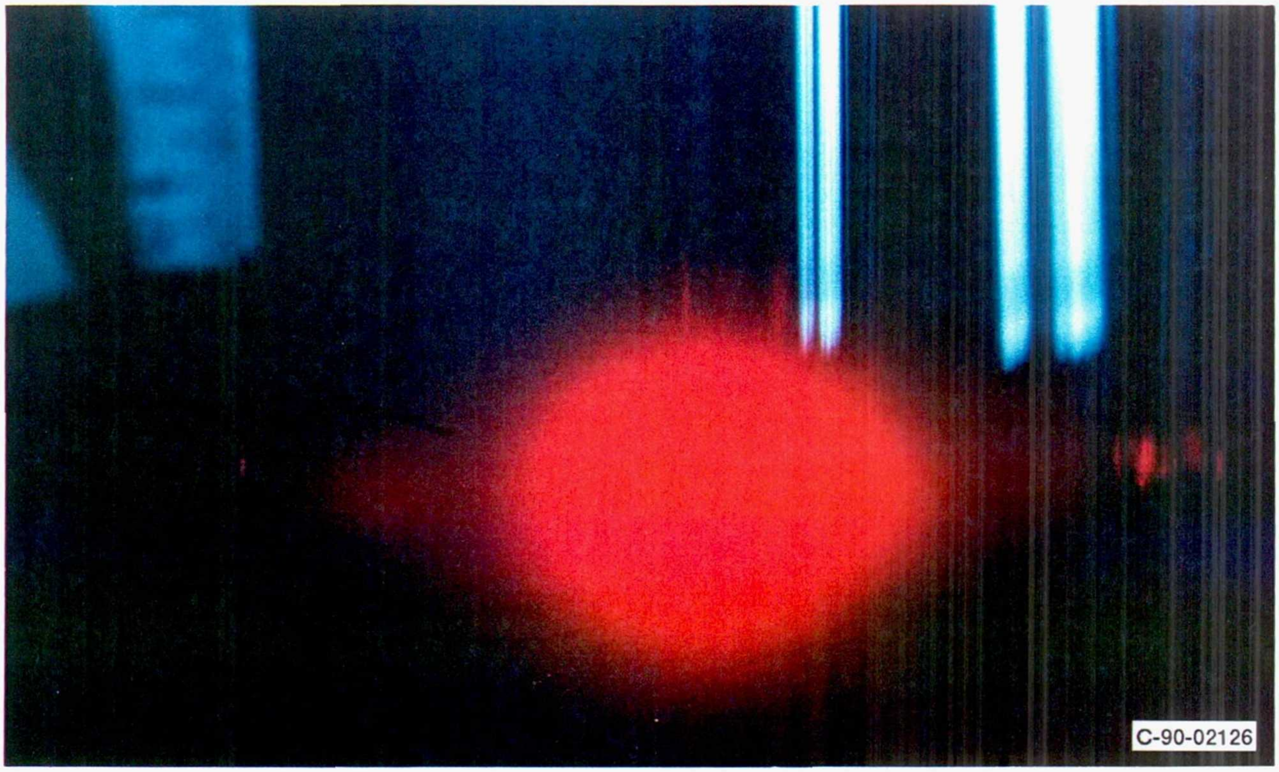

Figure 7.—Example spot size photograph.

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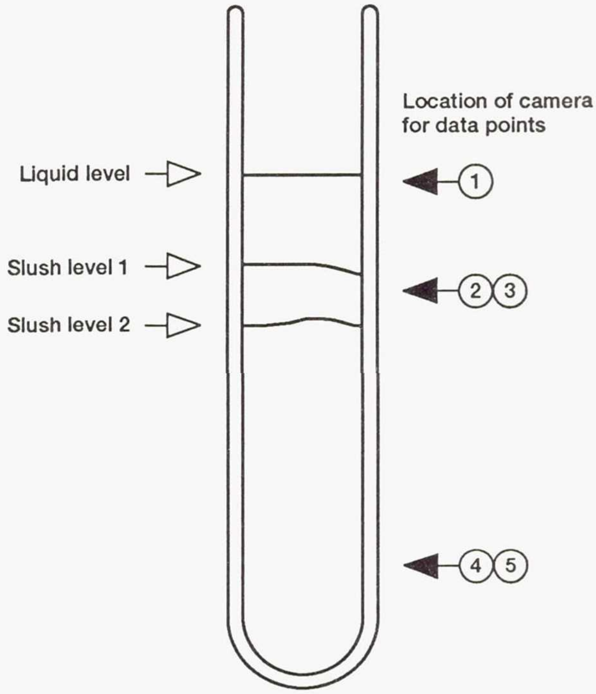

Figure 8.—Location of camera/laser for acquisition of the five spot size photographs.

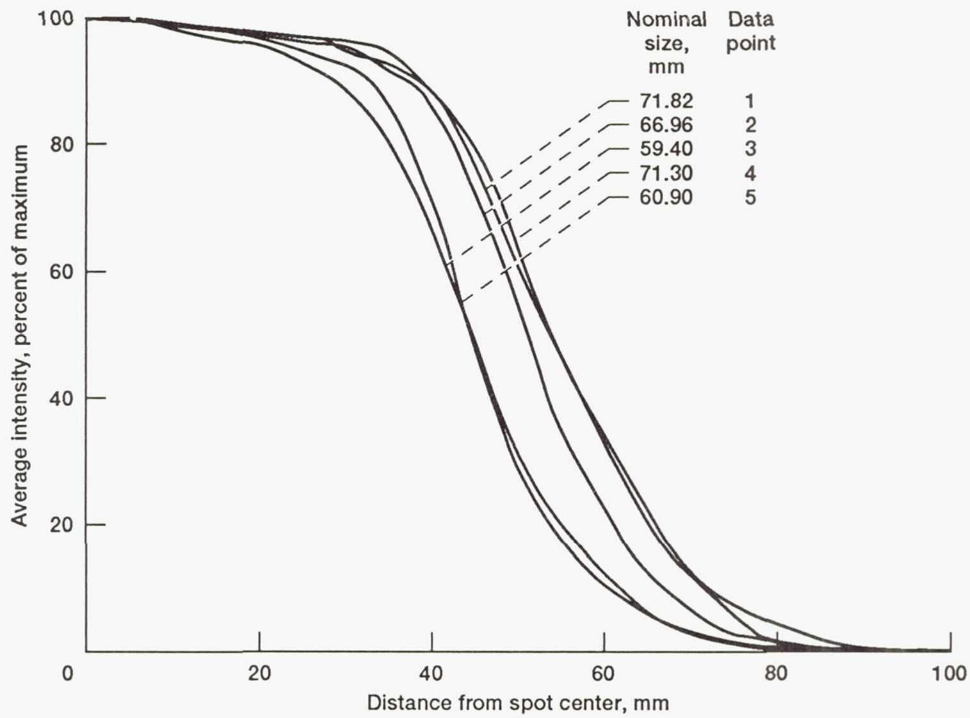

Figure 9.—Normalized spot radii of five configurations using Gaussian derived centers.

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY (*Leave blank*)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
		Technical Memorandum

4. TITLE AND SUBTITLE

An Optical Method for Determining Level in Two-Phase Cryogenic Fluids

5. FUNDING NUMBERS

WU-590-21-11

6. AUTHOR(S)

Lawrence G. Oberle and Donald H. Weikle

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135-3191

8. PERFORMING ORGANIZATION REPORT NUMBER

E-6398

9. SPONSORING/MONITORING AGENCY NAMES(S) AND ADDRESS(ES)

National Aeronautics and Space Administration
Washington, D.C. 20546-0001

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

NASA TM-104524

11. SUPPLEMENTARY NOTES

Responsible person, Lawrence G. Oberle, (216) 433-3647.

12a. DISTRIBUTION/AVAILABILITY STATEMENT

Unclassified - Unlimited
Subject Category 35

12b. DISTRIBUTION CODE

13. ABSTRACT ***(Maximum 200 words)***

A method has been evaluated to measure the liquid-gas and the liquid-slush interfaces in two-phase cryogen systems using optical means. This method makes use of the attenuation of a directed light beam caused by the difference in the index of refraction between the solid particles and the surrounding liquid. Preliminary experimental results obtained in slush nitrogen are shown. The possibility of extending this technique to include a measure of solid fraction is also discussed.

14. SUBJECT TERMS	15. NUMBER OF PAGES
Optics; Level detection; Slush; Cryogenics	14
	16. PRICE CODE
	A03

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	Unclassified	